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<b>(21) International Application Number:</b> PCT/US92/10002 <b>(22) International Filing Date:</b> 25 November 1992 (25.11.92)  <b>(30) Priority data:</b> 800,177 27 November 1991 (27.11.91) US  <b>(71) Applicant:</b> E.I. DU PONT DE NEMOURS AND COMPANY [US/US]; 1007 Market Street, Wilmington, DE 19898 (US).  <b>(72) Inventors:</b> FRANKOSKY, Michael, Stephen ; 1825 Graves Road, Hockessin, DE 19707 (US). KWOK, Wo, Kong ; 11 McCormic Drive, Hockessin, DE 19707 (US). ZIESEL, Donald, Titus ; 604 Northside Drive, Wilmington, DE 19809 (US).		<b>(74) Agents:</b> HIGGS, W. Victor et al.; E.I. du Pont de Nemours and Company, Legal/Patent Records Center, 1007 Market Street, Wilmington, DE 19898 (US).  <b>(81) Designated States:</b> JP, KR, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
<b>(54) Title:</b> NEW FIBERFILL BATTINGS  <b>(57) Abstract</b>  Improved polyester fiberfill batts for apparel and other uses are prepared of polyester fiber and binder fiber, sprayed with a soft resin by oven bonding and hot roll treatment. This provides bonded batting which is characterized by softness and drapability, good insulating performance, low levels of fiber leakage or percolation through shell fabrics, enhanced durability when laundered by washing/drying or by dry cleaning, and enhanced structural integrity whereby it hangs freely without the need for quilting into small size panels.		

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## TITLE

New Fiberfill Battings

## FIELD OF THE INVENTION

5           This invention is concerned with improvements  
in and relating to bonded polyester fiberfill batts,  
sometimes referred to as battings, especially processes  
whereby such improved batts with desirable aesthetic and  
serviceable qualities may be obtained, and to articles  
10           incorporating such improved batts.

## BACKGROUND OF THE INVENTION

          Polyester fiberfill (sometimes referred to as  
polyester fiberfilling material) is well accepted as a  
15           reasonably inexpensive filling and/or insulating material  
for pillows, cushions and other furnishing materials,  
including bedding materials, and in apparel, and is  
manufactured and used in large quantities commercially.  
For many of these uses, as disclosed, e.g., in U. S.  
20           Patents: Tolliver U.S. 3,772,137; Stanistreet U. S.  
4,068,036; Scott U. S. 4,129,675; Pamm U. S. 4,281,042;  
Frankosky U. S. 4,304,817; Siniscalchi U. S. 4,551,383;  
and LeVan U.S. 4,869,771, it has been desirable to make  
bonded batts, e.g., by spraying a resin-bonding agent,  
25           usually of an acrylic polymer, or by blending the  
polyester fiberfill with binder fibers, such as are well  
known in the art, or by use of both a resin-bonding agent  
and binder fibers.

30           To improve the aesthetics of polyester  
fiberfill, it has often proved desirable to "slicken" the  
fiberfill with a coating of durable (i.e., wash-  
resistant) coating that has usually been a silicone,  
i.e., a cured polysiloxane as disclosed, e.g., by Hofmann  
U. S. Pat. No. 3,271,189; Mead et al U. S. Pat. No.  
3,454,422; Ryan U. S. Pat. No. 3,488,217; Salamon et al

U. S. Pat. 4,146,674; LeVan, above; Takemoto Oil and Fat Co., Ltd., Japanese Published Application No. 58-214,585(1983); or other types such as the polyalkylene oxide variety disclosed by, e.g., Marcus U.S. Patent No. 4,818,599.

Despite all the prior suggestions and commercially-available materials, especially for use in premium level apparel products, sleeping bags, and comforters, there still remains a need for an easily prepared, homogeneous batting that is characterized by softness and drapability to conform to the wearer's body, good insulating performance, low levels of fiber leakage through shell fabrics, enhanced durability to laundering by washing/drying or by dry cleaning, and enhanced structural integrity whereby the batting is able to hang freely without the need for having it quilted into small size panels.

#### SUMMARY OF THE INVENTION

According to one aspect of the invention, there is provided a process for preparing a bonded batt, comprising forming a blend of polyester fiberfill, in amount by weight about 70 to about 96%, intimately mixed with a binder fiber, preferably a bicomponent binder fiber, having binder material of melting point lower than the softening point of the polyester fiberfill, in amount by weight about 4 to about 30%, preparing a continuous batt from said blend, said batt having an upper face and a lower face, advancing said batt through one or more spray zones, whereby both faces of the batt are sprayed with resin, in total amount about 10 to about 30% of the weight of the sprayed batt, including the resin, said resin being selected to provide, after curing, a cured resin having a glass transition temperature (Tg) of about 0 degrees Celsius or less, heating the sprayed batt in an oven to cure the resin and soften the binder material, followed by hot-rolling the heated batt to achieve

intimate contact between the resin and the fibers in the faces of the batt, and cooling the rolled batt.

The hot rolling is preferably effected by use of heated rolls in a calender or S-wrap configuration.

5 According to another aspect of the invention, there is provided a bonded batt, comprising polyester fiberfill of 0.2 to 10 dtex per filament, bonded throughout with lower melting binder material (from the binder fiber used in the process) in amount by weight about 2 to about 25% of the weight of the batt, and with upper and lower faces of said batt being sealed with a resin having a glass transition temperature ( $T_g$ ) of about 10 0 degrees Celsius or less, in amount about 10 to about 30% of the weight of the batt, whereby the sealing rating (SR, as defined) of said faces is at least 3, said batt having a wash durability (WD, as defined) of at least 3, and a bending stiffness (B, as defined) of about 80 15 cN/cm<sup>2</sup> or less, preferably about 50 cN/cm<sup>2</sup> or less.

#### DETAILED DESCRIPTION OF THE INVENTION

20 Thus, the invention provides fiberfill batts, such as are needed for use in premium apparel, by first preparing a homogeneous blend of polyester fiberfill (70-96% by weight of the blend) and a suitable binder fiber (4-30% by weight of the blend). This blend is 25 converted on a card or garnet to a web which may then be layered or cross lapped to form a batting to whose upper and lower faces is serially applied a suitable latex (e.g., a colloidal dispersion of acrylic polymers and/or copolymers in water, discussed in more detail herein- 30 after), e.g., by spraying. The sprayed batting is heated, e.g., conveniently by being passed through a heated oven to dry the coating(s) and to polymerize the polymeric component(s) to high molecular weight, and to activate the binder fiber. This may be conveniently done in three passes through such an oven, two to serially cure each coating, after such coating is applied to each face, and

a third pass to supplement the other two and to activate the binder fiber in preparation for the hot-rolling. The bonded batt is passed around or through heated rolls (S-wrap or calendering process) to soften and spread the cured resin and ensure its complete and even distribution among the fibers in the two faces (large surfaces) of the batt to prevent fiber leakage through the batt and, if needed, to ensure that the batt is of the desired thickness.

The resins that may be used herein are termed variously, by different manufacturers, as "soft" or "medium", or even "very soft", but are characterized by having second order glass transition temperatures ( $T_g$ ) of about 0 C or less. They provide both softness and drapability to the batt when used in, e.g., apparel, while acting as barrier to fiber leakage from the batt. The final batts may have a basis weight of 1.5 to 12 oz./yd<sup>2</sup>. (50 to 400 g./m.<sup>2</sup>) and a thickness of 0.07 to 0.20 inch/oz./yd.<sup>2</sup> (0.05 to 0.15 mm./g./m<sup>2</sup>). Thus the batts of this invention are prepared from a blend of polyester fiberfill and binder fibers, and the fibers in the faces are sealed by a suitably soft-type resin coating. The polyester fiberfill may all be slickened, e.g., as described herein, or may be blend of slickened and unslickened fibers. The fiberfill may be solid, hollow, or a blend of solid and hollow fibers and is not limited to any type of fiber cross section, i.e., it may be of cruciform, trilobal, Y-shaped, dog bone, scalloped oval, and other non-circular cross sections as well as round. The fiberfill has a denier per filament (dpf) within the range of 0.2 to 10, with a dpf of about 1.65 being singularly preferred, and constitutes about 70 to 96% by weight of the blend. The individual fibers are provided with crimp by conventional means and typically exhibit from 5 to 15 crimps per inch and have a length within the range of 3/4 to 3 inches. The binder fibers

constitute from about 4 to 30% by weight of the batt and may be of the sheath/core (s/c), side/side (s/s), or monocomponent types. These may be obtained from (co)polyesters, polyolefins, polyolefin/polyester, polyamide/polyamide, e.g., and the like. Useful types of binder fibers, and their modes of functioning, are described in, e.g., "Nonwovens World", March/April, 1990, page 37. The initial dpf of suitable binder fibers in the blend is typically within the range of 2 to 15 with a dpf of 4 being commonly used. Useful binder fibers include those disclosed in the aforementioned U. S. Patents to Scott, Pamm, Frankosky, and Marcus, together with those shown in Harris et al U. S. Patent No. 4,732,809; Taniguchi et al U. S. Patent No. 4,789,592; Tomioka et al U. S. Patent No. 4,500,384; Hirose et al Japanese Patent Publication Kokai 57-210,015(1982); and others known in the art which will function within the oven temperatures disclosed herein. Preferred binder fibers include the commercially-available "Melty 4080" (Unitika Co., Japan) and the "ES" and "EA" polyolefins (Chisso Corporation, Japan).

The cured resin coating on the batt constitutes about 10 to 30% by weight of the final bonded batt, with 12 to 25% being preferred, and about 18% being singularly preferred. As noted previously, a suitable resin coating has a Tg of about 0 C or less. The useful resins are obtained from commercially-available acrylic and vinyl latex compositions among which are included, e.g., Rhoplex E-32 (Rohm and Haas Co.), TR-934 (Rohm and Haas Co.), X-4280J (Kanebo, Japan), these Hycar® latex compositions of B. F. Goodrich Co.: 26146, 26171, 26322, 26083, 26092, 2671, 26120, 2679, 26796, these latex products of National Starch and Chemical Corporation: NACRYLIC X 4445, NACRYLIC X 788-6007, NACRYLIC X 4483, NACRYLIC X 4460, NACRYLIC X 4260, NACRYLIC X 4425, NACRYLIC X 4465, NACRYLIC 4401, NACRYLIC X 78-3990,

NACRYLIC X 78-3997, NACRYLIC X 78-3905, NACRYLIC X 4280, NACRYLIC 4441, NACRYLIC 78-6114, X-LINK 2873, X-LINK 2849, X-LINK 78-6119, X-LINK 2893, X-LINK 2833, X-LINK 78-6004, X-LINK 2813, RESYN 2375, DUR-O-SET E-230, DUR-O-SET E-669, and other commercially-available latexes which are cured to resins whose Tg values are about 0 C or less. Some of such commercially-available resins and their Tg values are listed in brochures, e.g., one by B. F. Goodrich, dated 1989, entitled HYCAR® Acrylic Latexes, and one by National Starch and Chemical Corporation, entitled Binders, Saturants, Laminants.

Preparation of the batts is generally begun by conventional opening and blending of the polyester fiberfill and binder fiber, followed by carding or garnetting to make a web. This web can be layered with other webs from a train of cards or garnets, or it can be cross lapped and combined with other webs to form an unbonded batting. This batting is then sprayed with the latex composition on both sides of the batting and is fed to the oven for curing of the resin and bonding of the binder fibers. The oven treatment is conducted at 150-190 C for 2 to 5 minutes, and is conveniently done in three passes of the batt, as previously noted. The bonded batt is then passed through/around at least two hot rolls having a surface temperature in the range of 150 to 250 C (more than two rolls may be used). The configuration of the batting may be in S-wrap over the rolls to provide maximum contact with the rolls. The latter may have a clearance of from 2 to 5 mm. depending on the final batting thickness desired. Alternatively, the bonded batting may be passed through calender rolls, heated as above. In these treatments, only one roll may be heated, if desired, and the batt is passed through/over the rolls a second time to heat the opposite side of the batt. Contact time on the rolls is from 3 to 25 seconds. The hot roll treatment softens and spreads the resin to



ensure its complete and even distribution on the batt surface(s) to prevent fiber leakage and to provide a uniform surface, free of lumps, for comfort and aesthetic performance in use. The batts exhibit the basis weight and thickness ranges previously indicated.

The batts of this invention exhibit desirable levels of thermal resistance or insulation, commonly reported as CLO ratings (see Hwang U.S. Patent No. 4,514,455). Batts of this invention desirably exhibit a CLO value of at least about 0.36 CLO/ oz./yd.<sup>2</sup> and preferably 0.48 CLO/ oz./yd.<sup>2</sup> or higher.

It is to be understood that the components and processes described herein should be selected to provide the batts of this invention. Care must be taken to select combinations that do so provide. For example, the slickener on the fiber and the latex applied to the batt should be selected so as to adhere sufficiently, so that the final batt may exhibit, for example, sufficient wash durability.

#### TEST PROCEDURES

CLO ratings are obtained as described in Hwang, above.

Wash durability ("WD") of the batts of this invention is evaluated by the procedures of ASTM D-4770-88. In the Examples, the panels were 24 inches x 24 inches in size. Durability ratings are reported for measurements made according to paragraph 8.6.1. Batts of the invention exhibit a rating of 3 or higher (paragraph 8.5 scale).

Fiber leakage or percolation through shell fabric is measured as a sealing rating ("SR") by the method described in LeVan U. S. Patent No. 4,869,771, with a sealing rating (SR) of 5 being excellent and a sealing rating (SR) of 1 being poor. The batts of this invention exhibit a sealing rating (SR) of 3 or higher.

The softness or drapability of the batts of

this invention is measured according to German Industrial Standard 53362 Cantilever (DIN 53362 Cantilever) which determines and totals the bending stiffness ("B") of the batting in machine and cross machine directions; the combined results are related to drapability and softness. Batting samples are cut to 25 cm. length and 2.5 cm. width, and are cut in both machine (MD) and cross machine (XD) directions. Each Test specimen is weighed and its weight recorded as "W". Bend length ("LU") is then determined by sliding the sample horizontally on a platform until the front of the bent sample reaches an angle of 41 degrees and 30 seconds. The following calculation is then made:

$$B = F_1(LU \div 2)^3$$

where B = bending stiffness in  $\text{cN/cm}^2$

LU = bend length in cm.

$F_1 = 9.8 (W \div L)$

W = weight of the specimen sample in grams

L = sample specimen length in cm.

The batts of this invention exhibit a bending stiffness ("B" being the sum of values determined for MD and XD samples from the batt) of  $80 \text{ cN/cm}^2$  or less.

#### EXAMPLES

##### EXAMPLE 1

An 82 lb. sample of polyester staple containing 50 weight percent silicon-slickened fiber of 1.65 dpf and 2 inch cut length and 50 weight percent dry (no slickener) fiber of the same denier and cut length was opened by a conventional mechanical opener and fed to a hopper. In a separate opener was placed 18 lb. of "Melly 4080" binder fiber (4 dpf, 2 inch cut length, 50/50 s/c) which had been pre-opened. The binder fiber was fed to the same hopper containing the staple blend and the fibers were mixed, first by hand, then by mechanical

tumbling of the combined actions of the inclined and horizontal aprons.

5           The mixed fibers were fed to two separate  
garnets which each produced a continuous web about 60  
inches wide and having a basis weight of about 1  
oz./yd.<sup>2</sup> (34 g/m.<sup>2</sup>). Each web was passed through a  
separate cross lapper which produced a cross lapped batt  
which was placed on a moving conveyor whose speed was  
10       about 8 yd./min (7.3 m./min.). The conveyor collected and  
combined both cross lapped batts into a final multiple-  
layered batt having a basis weight of about  
2.7 oz./yd.<sup>2</sup> (90 g./m.<sup>2</sup>). In a continuous operation, this  
batt was passed into a spray zone where Kanebo's X-4280J  
15       latex was applied to the top side of the batt which was  
then passed into a 3-path oven (sufficient latex was  
applied to provide 9% by weight cured resin on the batt).  
This path was at 150 C and the resin was cured and the  
binder fiber activated during a residence time of about 1  
20       minute in the oven. After the batt exited the oven, it  
was inverted, latex applied to the top side ("new") of the  
batt, and the batt was carried by a second conveyor to a  
second path of the oven (170 C) to cure the resin and  
activate the binder fiber (resin at 9% by weight resulted  
25       on this side of the batt to make a total of 18% by weight  
resin on the batt). The batting was fed to the third path  
of the oven (170 C) to provide further heating of the  
batt for an additional minute (total heating is for 3  
minutes).

30           The bonded batt is passed through a pair of hot  
rolls in S-wrap configuration (roll surfaces at 200 C),  
with a roll contact time of about 12 seconds; roll  
separation was 2 mm. The batting is compressed to about  
one half its original thickness and is wound up into a  
roll. This batting (18% resin, 18% binder fiber) had a  
basis weight of 3.33 oz/yd.<sup>2</sup>, a thickness of 0.41 inch,

exhibited a wash durability rating of 4, a sealing rating of 5, and total bending stiffness of 22.1 cN/cm.<sup>2</sup> (MD = 8.6, XD = 13.5).

#### EXAMPLE 2

In the following Table are reported the properties of other batts of the invention, prepared by the apparatus and processes described in Example 1, above, using the same latex, oven and roll temperatures and times as in Example 1. In the Table, "Fiber A" is the fiber blend of Example 1. In all other indicated "Fibers" ("B", etc.), the binder fiber ("Melly 4080") had already been combined with the fiberfill and was not separately added as shown in Example 1.

TABLE

ITEM NO.	BATTING			BASIS		B				
	FIBER	BINDER	RESIN	WEIGHT (OZ/YD2)	THICKNESS (INCHES)	WD	SR	MD	CD	TOTAL
1	A	18	25	3.14	0.41	4	5	33.5	35.6	69.1
2	A	25	18	2.86	0.35	4	5	20.1	31.1	51.2
3	B	22	12	2.76	0.35	4	5	23.1	38.1	61.2
4	C	15	18	3.24	0.31	5	5	14.9	18.8	33.7
5	D	25	18	3.08	0.33	4	5	13.2	34.6	47.8

Where Fiber B is a 78/22 (W/W) blend of (1) 5 dtex, solid, round cross-section, 50 mm cut length, polyethylene terephthalate staple bearing a polyalkylene oxideslickener and (2) "Melly 4080" (4 dpf);

Fiber C is a 78/7/15 (W/W/W) blend of (1) solid, round cross-section, silicone-slickened, 3 dpf polyethylene terephthalate staple, (2) 7-hole hollow round cross-section, silicone-slickened, 5.5 dpf polyethylene terephthalate staple, and (3) "Melly 4080" (4 dpf); and

Fiber D is a 75/25 (W/W) blend of (1) 1.65 dpf solid, round cross-section, silicone-slickened, 2 inch cut length polyethylene terephthalate staple and (2) "Melt 4080" (4 dpf).

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What we claim is:

1. A process for preparing a bonded batt, comprising forming a blend of polyester fiberfill, in amount by weight about 70 to about 96%, intimately mixed with a binder fiber having binder material of melting point lower than the softening point of the polyester fiberfill, in amount by weight about 4 to about 30%, preparing a continuous batt from said blend, said batt having an upper face and a lower face, advancing said batt through a spray zone, whereby both faces of the batt are sprayed with resin, in total amount about 10 to about 30% of the weight of the sprayed batt, including the resin, said resin being selected to provide, after curing, a cured resin having a glass transition temperature (Tg) of about 0 degrees Celsius or less, heating the sprayed batt in an oven to cure the resin and soften the binder material, followed by hot-rolling the heated batt to achieve intimate contact between the resin and the fibers in the faces of the batt, and cooling the rolled batt.

2. A process according to Claim 1, wherein the hot-rolling of the heated batt is effected by passing the batt between heated rolls in a calender.

3. A process according to Claim 1, wherein the hot-rolling of the heated batt is effected by passing the batt around heated S-wrap rolls.

4. A bonded batt, comprising polyester fiberfill of 0.2 to 10 dtex per filament, bonded throughout with lower melting binder material in amount by weight about 2 to about 25% of the weight of the batt, and with upper and lower faces of said batt being sealed with a resin having a glass transition temperature (Tg) of about 0 degrees Celsius or less, in amount about 10 to about 30% of the weight of the batt, whereby the sealing rating (SR, as defined) of said faces is at least 3, said

batt having a wash durability (WD, as defined) of at least 3, and a bending stiffness (B, as defined) of about 80 cN/cm<sup>2</sup> or less.

5           5.    A batt according to Claim 4, wherein the bending stiffness is about 50 cN/cm<sup>2</sup> or less.

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## INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 92/10002

<b>I. CLASSIFICATION OF SUBJECT MATTER</b> (if several classification symbols apply, indicate all) <sup>6</sup>		
According to International Patent Classification (IPC) or to both National Classification and IPC		
Int.Cl. 5 D04H1/64; D04H1/58		
<b>II. FIELDS SEARCHED</b>		
Minimum Documentation Searched <sup>7</sup>		
Classification System	Classification Symbols	
Int.Cl. 5	D04H	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched <sup>8</sup>		
<b>III. DOCUMENTS CONSIDERED TO BE RELEVANT<sup>9</sup></b>		
Category <sup>10</sup>	Citation of Document, <sup>11</sup> with indication, where appropriate, of the relevant passages <sup>12</sup>	Relevant to Claim No. <sup>13</sup>
Y	EP,A,0 314 433 (E.I.DU PONT DE NEMOURS) 3 May 1989 cited in the application see the whole document ---	1
Y	EP,A,0 437 268 (NATIONAL STARCH) 17 July 1991 see abstract; claims 1,3,14,15,19; examples ---	1, 4
Y	US,A,3 772 137 (JAMES W. TOLLIVER) 13 November 1973 cited in the application see abstract; claims 1,2; examples ---	4
A	EP,A,0 265 221 (E.I. DU PONT DE NEMOURS) 27 April 1988 see abstract; claims 1,2,4,5-7,10 ---	1, 4
-/--		
<p><sup>10</sup> Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"A" document member of the same patent family</p>		
<b>IV. CERTIFICATION</b>		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
18 MARCH 1993	07. 04. 93	
International Searching Authority	Signature of Authorized Officer	
EUROPEAN PATENT OFFICE	DURAND F.C.	



III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claims No.
A	US,A,4 040 371 (JOHN LAMONTE COOPER) 9 August 1977 see the whole document ----	1,4
A	US,A,4 129 675 (PAUL T. SCOTT) 12 December 1978 cited in the application see abstract; claims 1-4; figures -----	1,2,4

**ANNEX TO THE INTERNATIONAL SEARCH REPORT  
ON INTERNATIONAL PATENT APPLICATION NO.**

US 9210002  
SA 67969

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.  
The members are as contained in the European Patent Office EDP file on  
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

18/03/93

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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US-A-4040371	09-08-77	BE-A- 852968 CA-A- 1068092 DE-A,B,C 2713851 FR-A,B 2346487 GB-A- 1572848 JP-C- 1092459 JP-A- 52120063 JP-B- 55030875 NL-A- 7703333 SE-B- 439003 SE-A- 7703547	29-09-77 18-12-79 06-10-77 28-10-77 06-08-80 16-04-82 08-10-77 14-08-80 03-10-77 28-05-85 30-09-77
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